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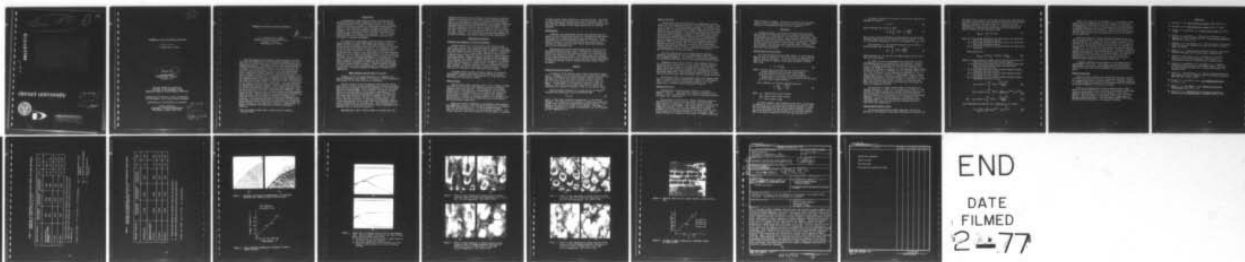
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TOUGHNESS OF NICKEL-MOLYBDENUM COMPOSITES.(U)  
FEB 75 S J BURDEN, A LAWLEY

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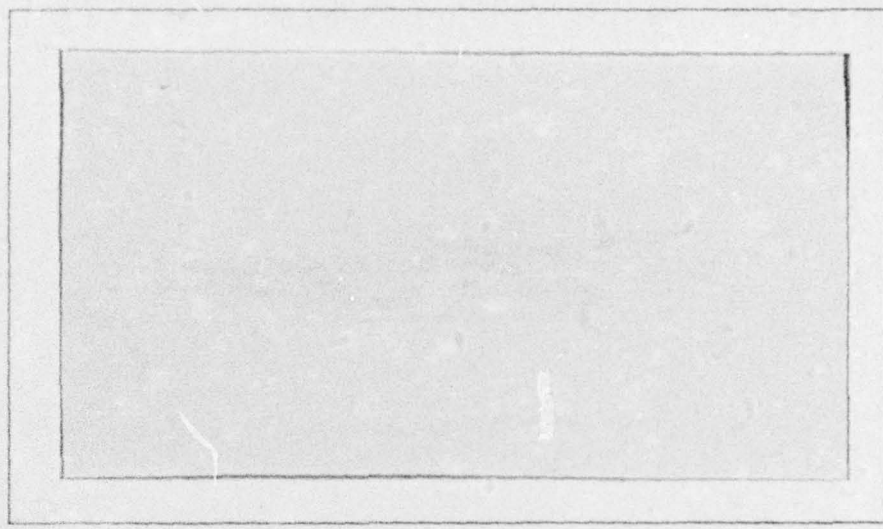
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TOUGHNESS OF NICKEL-MOLYBDENUM COMPOSITES

S. J. Burden and A. Lawley

February 1975

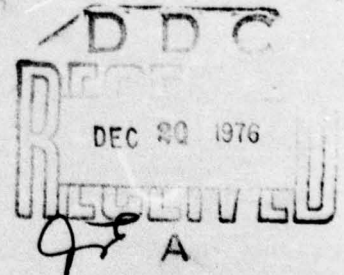
Technical Report  
Office of Naval Research  
Arlington, Va.

Contract #N00014-67-A-0406-0001  
(THE ROLE OF THE INTERFACE ON THE  
MECHANICAL BEHAVIOR OF METAL-MATRIX COMPOSITES)

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The correlation between toughness, microstructure, and interface form was examined for nickel-molybdenum composites ( $V_f$  0.08 and 0.20) in the diffusion-bonded condition and following elevated-temperature exposure. Instrumented Charpy tests were conducted on subsize specimens notched perpendicular to the direction of reinforcement. By testing above (ambient) and below the ductile to brittle transition temperature (DBTT) of the wire reinforcement, a direct comparison of impact response of ductile matrix-ductile fiber and ductile matrix-brittle fiber systems was possible. Composites exhibited a high level of toughness for all combinations of  $V_f$ , exposure history, and impact temperature; Charpy values were in the range 28-36 ft.lbs. (39-48.7J) and 10-19 ft.lbs. (13.5-25.7J) for  $V_f$  levels of 0.08 and 0.20, respectively. For each combination of test temperature and composite structure, increasing the level of reinforcement lowered toughness. Wire necking occurred at both  $V_f$  levels in the as-fabricated condition at room temperature. Pull-out was restricted to the lower volume fraction composites. Multiple fiber fracturing was a characteristic feature of low-temperature impact testing. Impact energy for the ductile matrix-brittle fiber condition can be predicted from the Cooper-Kelly model if an effective flow-stress is used for the matrix to accommodate work-hardening. A micromechanical model was developed for the ductile fiber condition which allowed for prediction of impact energy from tensile behavior of the constituents, and which accounted for the change in toughness associated with interface intermetallic compounds.

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## Introduction

A combination of high specific modulus and high specific strength make metal-matrix composites attractive for aircraft structural elements or in gas or steam turbines. To be viable, typical in-service conditions of loading also require reasonable levels of creep resistance, fatigue strength, and toughness. Presently only trends in toughness, measured in terms of impact response, can be predicted; there is no model with which to 'a priori' estimate the toughness of a composite. Only in systems reinforced with brittle fibers has the work of fracture been attributed to specific mechanisms.

The objective of the present study has been to characterize impact response in a model high-temperature composite, and to develop an understanding of the relationship between composite microstructure and toughness with particular reference to the role of the interface and interface structure. The composite system chosen for study consisted of a nickel matrix reinforced with molybdenum wires; alloys of the constituents are current candidate materials for gas turbine engine applications. The system has the advantage that, by examining toughness above and below the ductile-to-brittle transition temperature (DBTT) of the molybdenum, a direct comparison is possible between ductile fiber and brittle fiber-reinforced composites for invariant matrix properties and interface structure and/or strength. Primary variables included in the study were level of reinforcement  $V_f$ , extent of interface reaction due to elevated temperature exposure, and impact test temperature.

## Impact Behavior and the Work of Fracture

Possible sources of energy absorption in a composite are plastic deformation and fracture of the matrix, plastic deformation and fracture of the fiber, pull-out or debonding.

Briefly, Cottrell (1,2) has examined fiber pull-out and has shown that if this is the only energy absorbing mechanism operative, then the work of fracture should be proportional to  $V_f$ , the volume fraction of reinforcement. For a composite of brittle continuous fibers in a ductile matrix in which plastic deformation of the matrix is the energy absorbing mechanism, Cooper and Kelly (3,4) have shown that the work of fracture is dependent on  $(1-V_f)^2/V_f$  and  $U$ , the work of fracture per unit of volume of matrix. For fibers with some ductility, if the energy of debonding is equal to the strain energy stored in the fiber after debonding, then the work of debonding can be calculated (5). This has been shown to be much smaller than the work of pull-out, hence debonding will be important only if there is no pull-out.

Experimentally, only a limited number of studies have been

concerned specifically with toughness in metal-matrix 'synthetic' composites utilizing refractory metal wires for reinforcement (3, 4, 6-9). In the particular case of a nickel-alloy matrix, Winsa and Petrusek (9) measured impact response for a tungsten-reinforced superalloy above and below the DBTT of the reinforcement. Above the DBTT, toughness increased with increasing  $V_f$  since the reinforcement was tougher than the matrix; below the DBTT the reverse was true. Fiber pull-out and debonding did not play a major role in impact behavior. A modified rule-of-mixtures relationship was arrived at, based on the work of fracture of the constituents.

### Experimental Procedure

#### Materials and Composite Fabrication/Treatment

Matrix material was in the form of 0.010" (0.25mm) thick nickel sheet (Nickel 200; International Nickel Company 99.4%) and the reinforcement as fully annealed 0.010" (0.25mm) dia. molybdenum wire (KW Molybdenum; General Electric Company; 99.9%). Composites 2 1/4" x 1 1/2" x 5/16" (57mm x 38mm x 8mm) were fabricated by diffusion bonding between TZM dies in a vacuum hot press utilizing induction heating. Optimum processing parameters were determined to be 8000 psi (55.2 MN/m<sup>2</sup>) at 1000°C (1273°K) for 1 hour (3600s); this eliminated the original nickel-nickel interfaces. The nickel sheets were pre-grooved to a depth  $\sim 0.004$ " (0.1mm) to provide for wire alignment. Powdered boron nitride was a satisfactory parting agent between each die and the outer composite surfaces. Volume fractions of 0.08 and 0.2 were fabricated in this manner.

To assess microstructural stability, some of the composites were sealed in quartz tubes under a vacuum  $< 10^{-4}$  torr and then annealed at 1100°C (1373°K) for periods of 25, 50 and 100 hours ( $9 \times 10^4$ s,  $18 \times 10^4$ s and  $36 \times 10^4$ s).

#### Impact Testing

Subsize Charpy impact specimens (1/4" x 1/4"; 6.35mm x 6.35mm) were cut from the as-fabricated composite plates or following elevated temperature exposure. These blanks were then notched perpendicular to the direction of reinforcement, the notch being either parallel or perpendicular to the plane of the original nickel-nickel interfaces. In order to prevent fiber damage, it was necessary to perform all machining operations by grinding. For volume fractions of 0.08 and 0.2 there were 65 and 150 wires in the cross-section respectively.

Tests were run on a standard size instrumented Charpy machine. Low temperature ( $-196^\circ\text{C}$ : 77°K) tests were carried out in accordance with ASTM specification E23-72. In the instrumented Charpy test (10) the data, displayed on an oscilloscope, consist of: the striker velocity; load (force) applied to the specimen as a function



of time; and the energy absorbed as a function of time. The latter is the integration of the load versus time ( $\equiv$  distance) trace. It was found that energy readings from the oscilloscope and those taken from the dial readings of the Charpy machine were in excellent agreement.

### Metallography

Transverse and longitudinal sections were prepared from composites in the as-fabricated condition or following elevated temperature exposure. Standard procedures were followed plus a final treatment by vibratory polishing to ensure flat surfaces. Differential etching vis a vis the constituents was possible using a solution of copper sulfate and HCl for the matrix and Murakami's etch for the molybdenum wire.

Metallographic sections were examined either optically or by scanning electron microscopy (SEM). Intermetallic thicknesses at matrix-wire interfaces were measured by means of a calibrated eyepiece in the optical microscope. Interfacial areas were analyzed by the energy-dispersive probe in the scanning electron microscope. Fracture surface structures of Charpy test pieces were characterized by SEM; these surfaces could be examined directly without further treatment.

### Results

#### Elevated Temperature Exposure

On exposure, three distinct layers form at wire-matrix interfaces. A comparison of the interface in the as-fabricated composite and that following the most severe exposure condition studied, (100 hours at 1100°C;  $36 \times 10^4$ s at 1373°K) is made in Figure 1. The layers have tentatively been identified as MoNi, MoNi<sub>3</sub> and MoNi<sub>4</sub>. Time-dependence of the interface reaction is given in Figure 2. After 100 hours at 1100°C ( $36 \times 10^4$ s at 1373°K) the unreacted wire diameter is reduced to ~60% of its original value.

Since interface thickness is a linear function of (time)<sup>1/2</sup> a growth rate coefficient k can be determined from:

$$x^2 = kt \quad (1)$$

where x is the total interface thickness and t is the time of exposure. The coefficient is an empirical constant that takes into account both the rates of diffusion and compound formation. From Figure 2, the value of k is  $5.89 \times 10^{-11}$  cm<sup>2</sup>/sec. The interdiffusion coefficient for nickel in molybdenum is  $\sim 2 \times 10^{-10}$  cm<sup>2</sup>/sec at this temperature (11).



### Impact Properties

Charpy impact data for both levels of reinforcement and test temperature are summarized in Table 1. Each value of energy listed is the average of a minimum of three specimens. No difference was found to exist in impact response between Charpy test pieces notched perpendicular to the plane of the original nickel-nickel interfaces, and those notched parallel to them; the values reported in Table 1 refer to a notch orientation parallel to the former nickel-nickel interfaces but perpendicular to the direction of reinforcement. At both levels of reinforcement, complete fracture did not occur for any combination of test temperature and elevated temperature exposure. Thus, the impact data represent lower bound values. Oscilloscope traces (energy and load versus time) reproduced in Figure 3 compare impact response at relatively high and low levels of toughness respectively. Time-to-peak-load is a measure of plastic deformation in the test piece.

For each combination of test temperature and composite condition, increasing the level of reinforcement from 0.08  $V_f$  to 0.2  $V_f$  lowered toughness. The toughness of the 0.08  $V_f$  composite was essentially the same as that of the unreinforced nickel matrix.

Toughness of the 0.08  $V_f$  composite varied between limits of 28 and 36 ft.lbs (39 and 48.7J). For a given exposure condition (and hence known thickness of intermetallics at the interface) impact values were higher at the lower test temperature. This is attributed to the increase in ductility of the nickel matrix with decreasing temperature. The as-fabricated condition (i.e. minimum interface reaction zone) gave the highest level of impact resistance.

At the higher level of reinforcement ( $V_f = 0.2$ ) maximum toughness was exhibited in the as-fabricated condition at room temperature. A small increase in toughness was gained at the lower temperature, following elevated-temperature exposure.

### Fracture Morphology

Representative fracture surfaces (SEM) are collated in Figures 4 through 7. These afford a comparison and guide to the effect of level of reinforcement ( $V_f$ ), extent of interface reaction, and impact test temperature.

Wire necking was restricted to ambient temperature impact in the as-fabricated condition for both levels of reinforcement. Fiber pull-out was observed in the 0.08  $V_f$  composite but not at the higher level of reinforcement. The primary effect of elevated-temperature exposure, vis a vis the appearance of fracture surfaces, was to minimize separation of the matrix from the wires by pull-away, and to promote break-up of the interface. Under these conditions the composites came closest to complete separation

into two pieces on impact. Multiple fracturing of the molybdenum wires, as seen in longitudinal sections, was a characteristic feature of low-temperature impact testing, Figure 8.

### Discussion

Previous theories of the work of fracture have dealt only with the matrix contribution and with estimates of the interface contribution (i.e. fiber pull-out or fiber-matrix debonding). Thus, in the molybdenum-nickel system, impact behavior below the ductile-to-brittle transition temperature (DBTT) of the wire can be analyzed in light of existing models of brittle-fiber reinforcement. Extension to predict the observed impact response under conditions of ductile-fiber reinforcement (i.e. above the DBTT of the molybdenum wire) may then be possible.

### Ductile Matrix-Brittle Fibers

For a ductile matrix-brittle fiber system, it can be assumed that the work of fracture of the fibers (wires) is negligible compared to that of the matrix. If the work of fracture associated with the interface is ignored, then Cooper and Kelly (3) have shown that:

$$W = 2X V_m U_m \quad (2)$$

where  $W$  is the work of fracture per unit area due solely to plastic deformation and fracture of the matrix  
 $U_m$  is the fracture energy per unit volume of matrix  
 $X$  is the extent of the zone of plastic deformation on either side of the fracture plane parallel to the wire axis  
 $V_m$  is the matrix volume fraction.

For a condition of no matrix work-hardening (3):

$$X = \frac{\sigma_{my}}{2\tau_{my}} r \frac{(1-V_f)}{V_f} \quad (3)$$

where  $\sigma_{my}$  = matrix yield stress (normal)  
 $\tau_{my}$  = matrix yield stress (shear)  
 $r$  = wire (fiber) radius

In nickel, however, work hardening occurs and it is necessary to replace  $\sigma_{my}$  by  $\bar{\sigma}$ , an average flow stress for the matrix. After all the fibers have fractured, matrix material adjacent to fibers will not have deformed as much as the matrix in general, so that  $\tau_{my}$  can be retained as an approximation of matrix shear strength. Thus,  $X$  is given by equation (3) but with  $\sigma_{my}$  replaced by  $\bar{\sigma}$ .



The plastic behavior of the matrix is such that stress-strain response is given by:

$$\sigma = K \epsilon^n \quad (4)$$

and the average flow stress is then:

$$\bar{\sigma} = \frac{1}{\epsilon} \int_0^{\epsilon} K \epsilon^n d\epsilon = \frac{1}{\epsilon} \frac{K \epsilon^{n+1}}{n+1} \quad (5)$$

Since K and n can be determined from the tensile log true stress-log true strain curves of the nickel matrix,  $\bar{\sigma}$  is known. In turn, X is determined from equation (3) if it is assumed that  $\sigma_{my} = 2\tau_{my}$ .

From equation (4), the fracture energy per unit volume of matrix material ( $U_m$ ) is given by:

$$U_m = \int_0^{\epsilon} K \epsilon^n d\epsilon = \frac{K \epsilon^{n+1}}{n+1} \quad (6)$$

From equations (2), (3) and (6), the impact energy per unit area (W) can be calculated.

At  $-196^\circ\text{C}$  ( $77^\circ\text{K}$ ) the molybdenum wires are brittle so that experimental data may be compared with this analysis. Values of K,  $\epsilon$ ,  $\bar{\sigma}$ , X and  $U_m$ , determined from the pertinent stress-strain curves, are summarized in Table 2. For  $V_f = 0.2$  and with these values of  $\bar{\sigma}$ , X and  $U_m$  in equation (2), the calculated impact energy is  $4385 \text{ in.lb/in}^2$  ( $77.2 \times 10^4 \text{ J/m}^2$ ); this is reduced to  $1025 \text{ in.lb/in}^2$  ( $18.1 \times 10^4 \text{ J/m}^2$ ) if work hardening is neglected i.e.  $\sigma_{my}$  is used in place of  $\bar{\sigma}$  to calculate X. Experimental values of impact energy from the Charpy test are compared to this calculated value of  $4385 \text{ in.lb/in}^2$  ( $77.2 \times 10^4 \text{ J/m}^2$ ) in Table 3. The level of agreement with the experimental values (based on the fractured area below the notch) is encouraging since these are lower bounds by virtue of incomplete fracture.

The difference in impact energy between as-fabricated and exposed composites at  $-196^\circ\text{C}$  ( $77^\circ\text{K}$ ) is also seen in Table 3. The increment in impact energy after elevated temperature exposure is the contribution of the interface to the work of fracture,  $W_I$ . Thus,  $W_I$  increases as the thickness of the interface reaction zone increases; i.e. as the volume fraction of intermetallic  $V_{IM}$  increases, Figure 9. This contribution should be the same at room temperature since the reaction zone (intermetallics) is brittle at both temperatures.

#### Ductile Matrix-Ductile Fibers:

For ductile matrix-ductile fiber composites, the work of fracture is that needed to plastically deform and to fracture both



the fibers and the matrix. The energy absorbed can be divided into two parts, that absorbed while the fibers and matrix deform together and that absorbed by the matrix after the fibers have failed (assuming the matrix fracture strain  $\epsilon_m$ , is greater than that of the fiber  $\epsilon_f$ ). Ignoring the contribution of the interface, the work of fracture of the composite is then:

$$W_{\text{Total}} = W_m + W_f + W_m', \quad (7)$$

where  $W_m$  is the energy absorbed by the matrix while the fibers and matrix are deforming together

$W_f$  is the energy absorbed by the fibers while the fibers and matrix are deforming together

$W_m'$  is the energy absorbed by the matrix after the fibers have fractured.

From equation (2):

$$W_{\text{Total}} = 2X'V_m U_m + 2X'V_f U_f + 2XV_m U_m', \quad (8)$$

where  $X'$  is the distance on either side of the fracture plane over which the fibers and matrix deform together

$X$  is the distance on either side of the fracture plane over which the matrix deforms after the fibers have fractured (equation (3))

$U_m$  is the energy per unit volume of matrix absorbed during elongation from  $\epsilon=0$  to  $\epsilon=\epsilon_f$ ,

$U_f$  is the energy per unit volume of fiber absorbed during elongation from  $\epsilon=0$  to  $\epsilon=\epsilon_f$ ,

$U_m'$  is the energy per unit volume of matrix absorbed during elongation from  $\epsilon=\epsilon_f$  to  $\epsilon=\epsilon_m$ ,

$$U_m \text{ is equal to } \int_0^{\epsilon_f} K\epsilon^n d\epsilon = \frac{K(\epsilon_f)^{n+1}}{(n+1)} \quad (9)$$

$$U_m' \text{ is equal to } \int_0^{\epsilon_m} K\epsilon^n d\epsilon = \frac{K}{(n+1)} [(\epsilon_m)^{n+1} - (\epsilon_f)^{n+1}] \quad (10)$$

$$\text{and } U_f \text{ is equal to } \int_0^{\epsilon_f} K\epsilon^n d\epsilon = \frac{K(\epsilon_f)^{n+1}}{(n+1)} \quad (11)$$

$X$  is calculated from equation (3), replacing  $\sigma_{my}$  by  $\bar{\sigma}$  where:

$$\bar{\sigma} = \frac{1}{\epsilon_m - \epsilon_f} \frac{K}{n+1} [(\epsilon_m)^{n+1} - (\epsilon_f)^{n+1}] \quad (12)$$

There is no direct way to determine  $X'$ . In the present study a value for this parameter was obtained by means of a slip line field analysis of the plastic hinge in a notched bar subjected to a bending moment (12); this is an approximation since the material is assumed homogeneous and isotropic. This notwithstanding, the analysis gave a value for  $X'$  of 0.14" (3.56mm) which was consistent with the experimentally observed magnitude of lateral contraction in the composites.

With this value of  $X'$  and the calculated parameters  $U_m$ ,  $U_m'$  and  $U_f$  (from equations (9), (10), and (11); Table 2), impact energy can be estimated. For composites in the exposed condition, it is necessary to use the experimental fracture strain of the composite ( $\epsilon_c$ ) in place of the fiber fracture strain ( $\epsilon_f$ ) in equations (9) through (11). The experimental and calculated values are compared in Table 4 for  $V_f = 0.2$  at room temperature. The level of agreement between the calculated impact energy and that observed (based on the fracture area below the notch) is considered good. The interface contribution to the work of fracture is not included and the assumption that  $\tau_{my}$  is the shear yield strength of the matrix (ideally plastic condition) contributes some error.

A similar comparison was not possible at the lower level of reinforcement ( $V_f = 0.08$ ). These impact test pieces exhibited a large amount of bending at both temperatures and the fractured area was only about one half the original cross-section below the notch.

#### Fracture Morphology

Extensive fiber fragmentation and multiple fracture at  $-196^\circ\text{C}$  ( $77^\circ\text{K}$ ) is a consequence of the wire being below the DBTT. There is an accompanying extensive interface break up in composites subjected to elevated temperature exposure; this phenomenon was not restricted to locations adjacent to the fracture surface.

Even at room temperature fracture surfaces showed a large amount of fiber fracturing and splitting, particularly at the higher level of reinforcement. The fracture surfaces of the 0.08  $V_f$  composite exhibited fiber pull-out while those at the 0.2  $V_f$  level did not. This is probably a consequence of the large amount of bending accompanying impact. The instrumented Charpy load traces were particularly sensitive to the amount of bending.



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TABLE 1. Impact Properties of Nickel-Molybdenum Composites

Specimen Condition	Ft-Lbs*	In-Lbs/In <sup>2</sup> **	Peak Load*** (lbs)	Time to Peak Load (10 <sup>-3</sup> s)	V <sub>f</sub>	Test Temperature
Diffusion Bonded Nickel Sheets	32	7680	520	1.7	0	RT
	37	8890	600	1.8	0	-196°C
<u>Composites</u> As-Fabricated	33	7930	580	1.5	.08	RT
	19	4500	510	0.4	.20	RT
	36	8620	620	1.0	.08	-196°C
	12	2880	500	0.4	.20	-196°C
Exposed at 1100°C 25 hours	31	7450	520	1.3	.08	RT
	10	2400	450	0.2	.20	RT
	31	7450	600	1.5	.08	-196°C
	13	3120	510	0.4	.20	-196°C
50 hours	29	6970	520	1.3	.08	RT
	11	2650	480	0.3	.20	RT
	30	7200	580	1.2	.08	-196°C
	13	3120	530	0.4	.20	-196°C
100 hours	28	6600	510	1.3	.08	RT
	12	2880	480	0.2	.20	RT
	32	7680	600	1.4	.08	-196°C
	14	3360	550	0.4	.20	-196°C

1 hour =  $36 \times 10^2$  s

1J = 0.74 ft.lbf = 8.88 inch.lbs.

1kg = 2.2 lb.

°K = °C + 273

\* Dial Reading

\*\* Based on Specimen Cross Section Below Notch

\*\*\* From Load-Time Trace

TABLE 2. CALCULATED VALUES OF IMPACT PARAMETERS FOR NICKEL-MOLYBDENUM COMPOSITES

Material	$\bar{\sigma}$ (psi)	K (psi)	n	$\epsilon_f$	U in-lbs/in <sup>3</sup>	X (in)
Nickel R.T.	54,000	120,000	0.41	0.33	18,750	0.060
Nickel -196°C	76,900	190,000	0.56	0.44	33,840	0.081
Molybdenum R.T.	147,400	230,000	0.19	0.24	35,380	-

$$U = \int_0^{\epsilon_f} K(\epsilon_f)^n d\epsilon \quad \bar{\sigma} = \frac{1}{\epsilon_f} \int_0^{\epsilon_f} K(\epsilon_f)^n d\epsilon$$

$$X = \frac{\bar{\sigma}}{\sigma_{my}} \cdot \frac{(1-V_f)}{V_f}$$

$$1N/m^2 = 14.5 \times 10^{-5} \text{ lbf/in}^2; \quad 1J = 8.88 \text{ inch.lbs}; \quad 1 \text{ inch} = 25.4\text{mm}; \quad ^\circ K = ^\circ C + 273.$$



TABLE 3. COMPARISON OF EXPERIMENTAL AND CALCULATED IMPACT ENERGY FOR NICKEL-MOLYBDENUM COMPOSITES AT  $-196^{\circ}\text{C}$  ( $77^{\circ}\text{K}$ );  $V_f = 0.2$

Specimen Condition	Experimental* (in-lbs/in <sup>2</sup> )	Experimental** (in-lbs/in <sup>2</sup> )	Calculated*** (in-lbs/in <sup>2</sup> )	$V_{IM}$	$V_{Mo}$	$W_I$
As-Fabricated Exposed at $1100^{\circ}\text{C}$ : 25 hours	2880	> 3030	4385	.01	.188	-
	3120	> 3280	4385	.12	.148	240
50 hours	3120	> 3670	4385	.14	.134	240
100 hours	3360	> 3730	4385	.18	.124	480

\* Based on Charpy Dial Reading and Specimen Cross-Section Below Notch

\*\* Based on Charpy Dial Reading and Actual Fractured Area Below Notch

\*\*\* Equation (2)

1 hour =  $36 \times 10^2 \text{s}$ ;  $^{\circ}\text{K} = ^{\circ}\text{C} + 273$ ;  $1J = 8.8 \text{ inch.lbs.}$ ;

$V_{IM}$  = Volume fraction inter-metallic

$V_{Mo}$  = Volume fraction molybdenum

$W_I$  = Work of fracture of interface.



TABLE 4. COMPARISON OF EXPERIMENTAL AND CALCULATED ROOM TEMPERATURE IMPACT ENERGY FOR NICKEL-MOLYBDENUM COMPOSITES.  $V_f = 0.2$

Specimen Condition	Experimental* (in-lbs/in <sup>2</sup> )	Experimental** (in-lbs/in <sup>2</sup> )	Calculated (in-lbs/in <sup>2</sup> )	$V_{IM}$	$V_{Mo}$
As-Fabricated	> 4560	6000	6250	.01	.188
<u>Exposed at 1100°C:</u>					
25 hours	> 2400	2820	3200	.12	.148
50 hours	> 2650	2930	3140	.14	.134
100 hours	> 2880	3030	3100	.18	.124

\* Based on Dial Reading and Specimen Cross-Section Below Notch

\*\* Based on Dial Reading and Actual Fractured Area Below Notch

1 hour =  $36 \times 10^2$ s; °K = °C + 273; 1J = 8.8 inch.lbs.

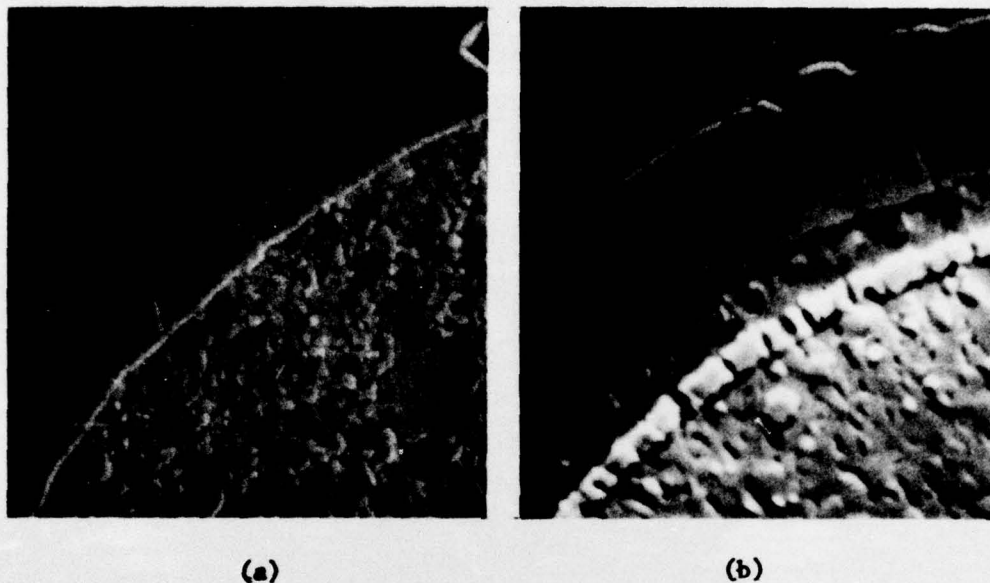


Figure 1. Interface structure (a) as-fabricated; (b) following 100 hours ( $36 \times 10^4$  s) at  $1100^\circ\text{C}$  ( $1373^\circ\text{K}$ ); X1000.

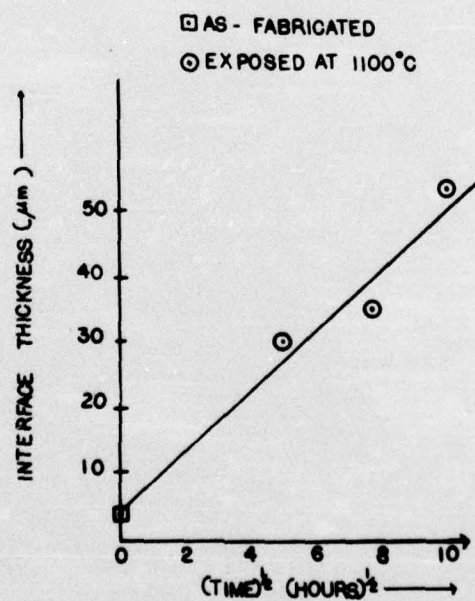
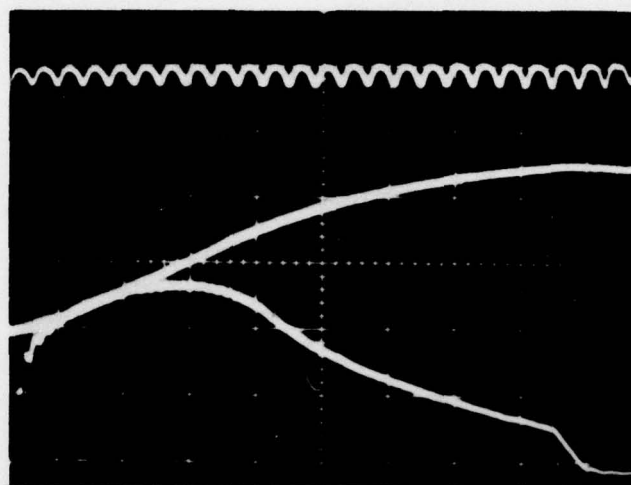
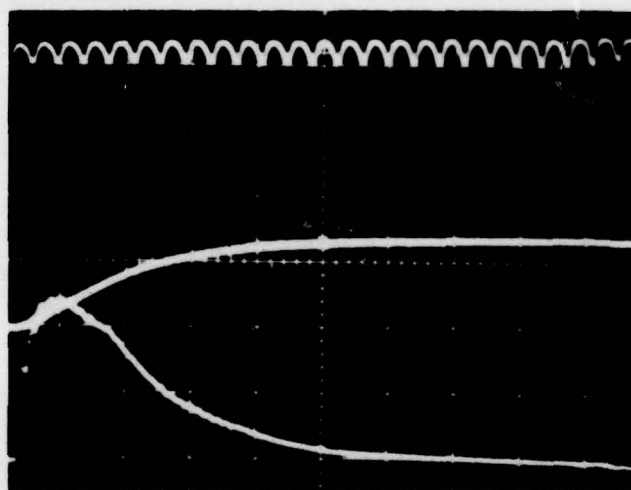


Figure 2. Total interface thickness as a function of time at  $1000^\circ\text{C}$  ( $1373^\circ\text{K}$ ).



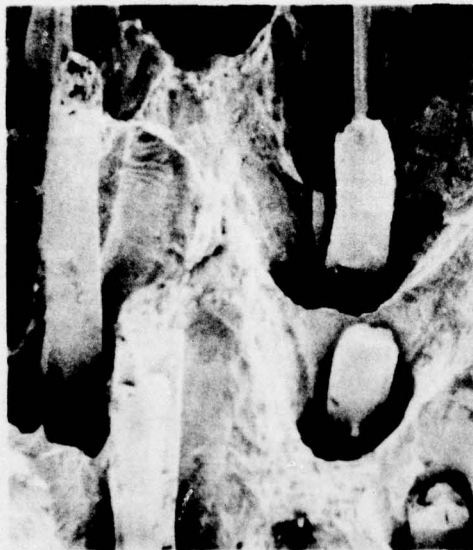


(a)



(b)

Figure 3. Traces from instrumented Charpy tests at room temperature. Time is  $0.5 \times 10^{-3}$  s per division and load ( $\equiv$  lower curve) 200 lbs (9100N) per division.  
 (a) as-fabricated,  $V_f = 0.08$ ; energy ( $\equiv$  upper curve) is 10 ft.lbs. (13.5J) per division.  
 (b) 100 hours ( $36 \times 10^4$  s) at  $1100^\circ\text{C}$  ( $1373^\circ\text{K}$ ),  $V_f = 0.2$ ; energy ( $\equiv$  upper curve) is 5 ft.lbs (6.75J) per division.



(a)



(b)

Figure 4. Effect of test temperature on Charpy fracture surface structure (SEM) of as-fabricated composites at  $V_f = 0.08$ ; X40 (a) room temperature; (b)  $-196^\circ\text{C}$  ( $77^\circ\text{K}$ ).



(a)



(b)

Figure 5. Effect of test temperature on Charpy fracture surface structure (SEM) of exposed composites (100 hours at  $1100^\circ\text{C}$ ;  $36 \times 10^4\text{s}$  at  $1373^\circ\text{K}$ ) at  $V_f = 0.08$ ; X40 (a) room temperature; (b)  $-196^\circ\text{C}$  ( $77^\circ\text{K}$ ).



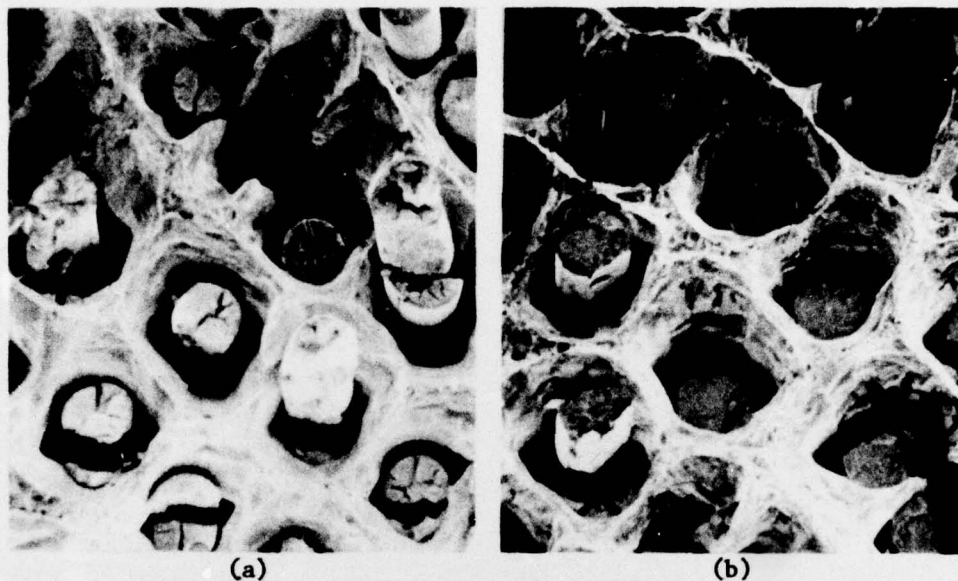


Figure 6. Effect of test temperature on Charpy fracture surface structure (SEM) of as-fabricated composites at  $V_f = 0.2$ ; X40 (a) room temperature; (b)  $-196^{\circ}\text{C}$  ( $77^{\circ}\text{K}$ ).

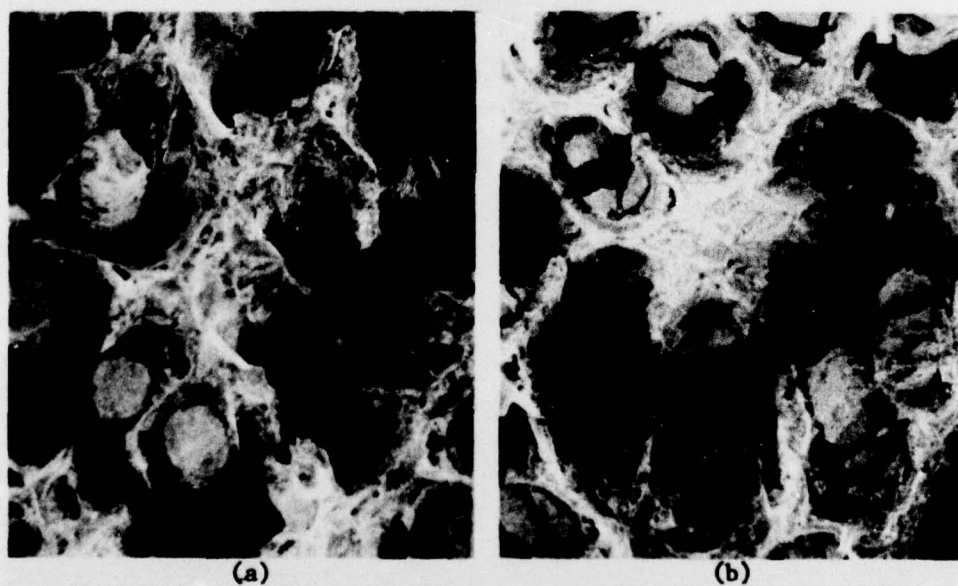


Figure 7. Effect of test temperature on Charpy fracture surface structure (SEM) of exposed composites (100 hours at  $1100^{\circ}\text{C}$ ;  $36 \times 10^4$  s at  $1373^{\circ}\text{K}$ ) at  $V_f = 0.2$ ; X40 (a) room temperature; (b)  $-196^{\circ}\text{C}$  ( $77^{\circ}\text{K}$ ).

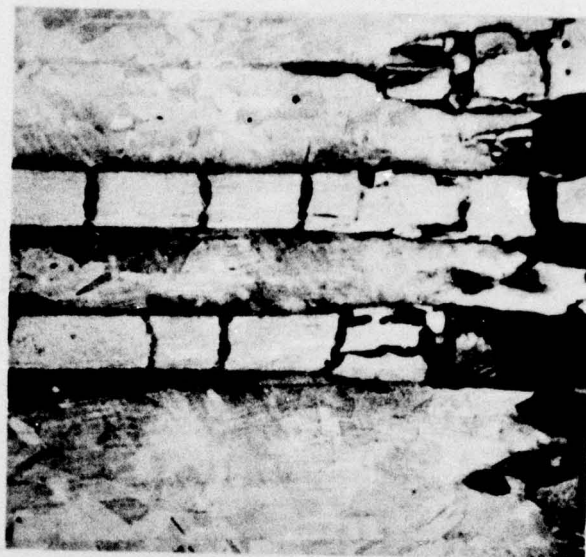


Figure 8. Multiple fiber fracture; impact tested at  $-196^{\circ}\text{C}$  ( $77^{\circ}\text{K}$ ); X25.

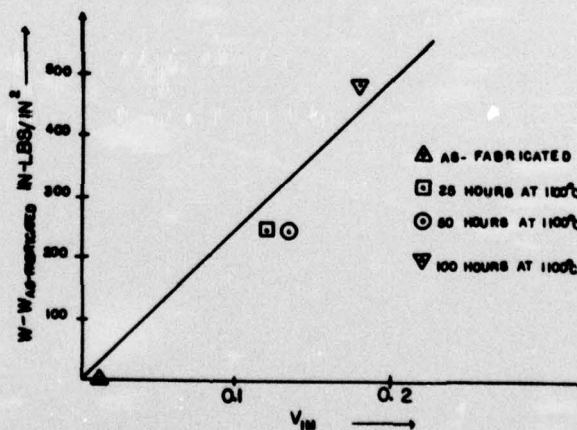


Figure 9. Increase in impact energy due to interface; tested at  $-196^{\circ}\text{C}$  ( $77^{\circ}\text{K}$ ).



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13. ABSTRACT The correlation between toughness, microstructure, and interface form was examined for nickel-molybdenum composites ( $V_f$ 0.08 and 0.20) in the diffusion-bonded condition and following elevated-temperature exposure. Instrumented Charpy tests were conducted on subsized specimens notched perpendicular to the direction of reinforcement. By testing above (ambient) and below the ductile to brittle transition temperature (DBTT) of the wire reinforcement, a direct comparison of impact response of ductile matrix-ductile fiber and ductile matrix-brittle fiber systems was possible. Composites exhibited a high level of toughness for all combinations of $V_f$ , exposure history, and impact temperature; Charpy values were in the range 28-36 ft.lbs. (39-48.7J) and 10-19 ft.lbs. (13.5-25.7J) for $V_f$ levels of 0.08 and 0.20, respectively. For each combination of test temperature and composite structure, increasing the level of reinforcement lowered toughness. Wire necking occurred at both $V_f$ levels in the as-fabricated condition at room temperature. Pull-out was restricted to the lower volume fraction composites. Multiple fiber fracturing was a characteristic feature of low-temperature impact testing. Impact energy for the ductile matrix-brittle fiber condition can be predicted from the Cooper-Kelly model if an effective flow-stress is used for the matrix to accommodate work-hardening. A micromechanical model was developed for the ductile fiber condition which allowed for prediction of impact energy from tensile behavior of the constituents, and which accounted for the change in toughness associated with interface intermetallic compounds.			

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